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IS 15304 (2003): Dipotassium Hydrogen Phosphate for Electroplating [CHD 5: Electroplating Chemicals and Photographic Materials]

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Indian Standard

DIPOTASSIUM HYDROGEN PHOSPHATE FOR ELECTROPLATING — SPECIFICATION

ICS 25.220.40,71.060.01

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

February 2003

Price Group 4

Inorganic Chemicals and Photographic Materials Sectional Committee, CHD 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by Inorganic Chemicals and Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

Dipotassium hydrogen phosphate (alternately known as potassium phosphate dibasic) (K_2HPO_4) is one of the important chemical used for electroplating with extensive usage for metal and gold plating.

There is no ISO/IEC Standard on the subject. This standard has been prepared based on practices prevalent in the field, in India.

The composition of the Committee responsible for formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

DIPOTASSIUM HYDROGEN PHOSPHATE FOR ELECTROPLATING — SPECIFICATION

1 SCOPE

This standard prescribes the requirements, methods of test and sampling for dipotassium hydrogen phosphate for electroplating.

2 REFERENCES

2.1 The standards listed below contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards.

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
265 : 1993	Hydrochloric acid (*third revision*)
266 : 1993	Sulphuric acid (*second revision*)
915 : 1975	One-mark volumetric flask (*first revision*)
1070 : 1992	Reagent grade water (*third revision*)
2088 : 1983	Method for determination of arsenic (*second revision*)
4905 : 1968	Method for random sampling

3 REQUIREMENTS

3.1 Description

Dipotassium hydrogen phosphate shall be in the form of colourless crystals somewhat hygroscopic but very soluble in water.

3.2 The material shall comply with the requirements prescribed in Table 1 when tested in accordance with the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 4 of Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed as agreed to between the purchaser and the supplier.

4.2 Marking

The container shall be marked with the following information:

a) Indication of source of manufacture,
b) Net mass of the material,
c) Name of the material,
d) Month and year of manufacture, and
e) Lot number or batch number.

Table 1 Requirements for Dipotassium Hydrogen Phosphate for Electroplating

(*Clause* 3.2)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. of Annex A)
(1)	(2)	(3)	(4)
i)	Dipotassium hydrogen phosphate content, percent by mass, *Min*	98.5	A-2
ii)	Water insoluble matter, percent by mass, *Max*	0.01	A-3
iii)	*p*H of 0.1M solution	9.3	A-4
iv)	Arsenic, percent by mass, *Max*	0.000 2	A-5
v)	Chloride, percent by mass, *Max*	0.003	A-6
vi)	Lead, percent by mass, *Max*	0.001	A-7
vii)	Sodium, percent by mass, *Max*	1.0	A-8
viii)	Sulphate, present by mass, *Max*	0.05	A-9
ix)	Mono or tribasic phosphate	To pass the test	A-10

4.2.1 *BIS Certification Marking*

The product may also be marked with the Standard Mark.

4.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 The method of preparing representative samples of the material and criteria for conformity with this standard shall be as prescribed in Annex B.

ANNEX A

(*Clause* 3.2 and *Table* 1)

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF DIPOTASSIUM HYDROGEN PHOSPHATE CONTENT

A-2.1 Reagents

A-2.1.1 *Standard Hydrochloric Acid* — 1 N.

A-2.1.2 *Methyl Red Indicator* — 0.1 g/100 ml alcohol.

A-2.2 Procedure

Weigh accurately about 4 g of the material, dry for 3 h at 105°C, and dissolve in 50 ml of water in a conical flask. Add 2 drops of methyl red, cool to room temperature and titrate with standard hydrochloric acid to a slight but distinct pink colour.

Calculate the content,
percent by mass = $(V \times 17.418)/M$

where

V = volume of 1N HCl used, and

M = of sample taken.

A-3 TEST FOR WATER INSOLUBLE MATTER

Weigh accurately 10 g of the material and dissolve in 100 ml water in a 250-ml beaker and heat on a steam bath for 1 h. Filter any undissolved residue, wash it with water, dry at 105°C. Cool in a desiccator, weigh and calculate as follows:

$$\text{Insoluble matter, percent by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = mass, in g, of residue; and

M = mass, in g, of the material taken for the test.

A-4 TEST FOR *p*H

Weigh accurately 1.74 g of the material and dissolve in 100 ml water in 250-ml breaker and determine the *p*H with a suitable *p*H meter.

A-5 TEST FOR ARSENIC

A-5.1 Reagents

A-5.1.1 *Concentrated Nitric Acid* — *See* IS 264.

A-5.1.2 *Concentrated Sulphuric Acid* — *See* IS 266.

A-5.2 Procedure

Weigh accurately about 1.0 g of the material. Transfer to an evaporating dish and dissolve in 10 ml of water. Add 5 ml of concentrated nitric acid and evaporate to dryness on a steam-bath. Re-evaporate with 10 ml of water and 5 ml of concentrated nitric acid. Take up the residue with 10 ml of water and 5 ml of concentrated sulphuric acid and evaporate to strong sulphuric fumes. Cool and then cautiously add 10 ml of water and proceed as prescribed in IS 2088 following the silver diethyl dithiocarbamate method.

A-6 TEST FOR CHLORIDE BY TURBIDIMETRIC METHOD

A-6.1 Apparatus

Spectrophotometer, UV-VIS wavelength range.

A-6.2 Reagents

A-6.2.1 *Dilute Nitric Acid* — 5 percent (*v/v*).

A-6.2.2 *Silver Nitrate* — 2.5 percent (*m/v*).

A-6.2.3 *Standard Chloride Solution* (1mg/ml)

Dissolve 1.648 g of sodium chloride (dried at 140°C for 1 h) in distilled water and dilute to 1 000 ml in a volumetric flask (1.00 ml = 1 mg Cl^-).

A-6.2.4 *Standard Chloride Solution* (0.01 mg/ml)

Dilute 10 ml of the standard stock solution (**A-6.2.3**) to 1 000 ml in a volumetric flask with distilled water.

A-6.3 Sample Solution

Dissolve 20 g of dipotassium hydrogen phosphate in distilled water and dilute to 200 ml in a volumetric flask.

A-6.4 Calibration Graph

Transfer 0, 2.0, 4.0, 6.0, 8.0 and 10.0 ml of the standard chloride solution (**A-6.2.4**) into six 50-ml volumetric flasks. Add 10 ml of 5 percent (*v/v*) nitric acid followed by 5 ml of 2.5 percent (*m/v*) silver nitrate and make upto 50 ml. Measure the absorbance of the solutions at 350 nm between 8 to 12 min using quartz cells against distilled water. Run the blank through the procedure and after deducting the blank absorbance plot the graph with the standards concentration *versus* absorbance. Verify the calibration by adding similar set of standards to same aliquots of the sample solutions (*see* Table 2).

A-6.5 Procedure

Take 20 ml of dipotassium hydrogen phosphate solution (**A-6.3**) into 50-ml volumetric flasks and carry out the procedure as described under **A-6.4** and record the absorbance against distilled water. Run the blank through the procedure and deduct its absorbance from the sample absorbance.

A-6.6 Calibration

$$\text{Chloride}\ (\text{Cl}^-)\ \text{in ppm} = [\,(A - B) \times 20\ \mu\text{g} \times C\,]/(D \times E)$$

where

A = absorbance of the sample,

B = absorbance of the blank,

C = dilution factor of the sample,

D = average absorbance of the standards, and

E = weight of the sample taken .

A-7 TEST FOR LEAD

A-7.1 Apparatus

A-7.1.1 *Nessler Cylinders* — Two, of 50-ml capacity.

A-7.2 Reagents

A-7.2.1 *Dilute Hydrochloric Acid* — approximately 1N.

A-7.2.2 *Hydrogen Sulphide Solution* — a freshly prepared, saturated aqueous solution of hydrogen sulphide gas.

Table 2 Absorbance for Standard Chloride Solution

(*Clause* A-6.4)

Standard Solution (1)	Absorbance (2)	Absorbance for 0.4 µg/ml (3)
Aqueous Standard Absorbance		
Reagent blank	0.005	
0.4 µg/ml	0.037	0.032
0.8 µg/ml	0.068	0.032
1.2 µg/ml	0,097	0.031
1.6 µg/ml	0.126	0.030
2.0 µg/ml	0.154	0,030
Sample + Standard Absorbance		
Reagent blank	0.005	
20 ml sample + 0.0 µg/ ml	0.008	
” + 0.4 µg/ml	0.038	0.030
” + 0.8 µg/ml	0.072	0.032
” + 1.2 µg/ml	0.099	0.030
” + 1.6 µg/ml	0.126	0.030
” + 2.0 µg/ml	0.153	0.029

The dipot hydrogen phosphate used in this experiment contains less than 5 ppm chloride.

A-7.2.3 *Standard Lead Solution*

Dissolve 0.160 g of lead nitrate in 100 ml of water containing 1 ml of concentrated nitric-acid and dilute with water up to the mark in a 1 000-ml volumetric flask. Dilute 10 ml of this solution to 100 ml in a 100-ml volumetric flask with water. One millimetre of this diluted solution is equivalent to 0.01 mg of lead (as Pb).

A-7.3 Procedure

Weigh accurately 2.00 g of the material, dissolve in 30 ml of water and dilute up to the mark with water in a 50-ml volumetric flask. Transfer 30 ml of this solution to Nessler cylinder A and 10 ml to Nessler cylinder B. Add 4 ml of the standard lead solution to Nessler cylinder B. Then add 1 ml of dilute hydrochloric acid followed by 10 ml of hydrogen sulphide solution to each of the cylinders and dilute to the mark with water.

A-7.3.1 The material shall be taken as having satisfied the requirement of the test if the solution in Nessler cylinder A is not darker than that in cylinder B.

A-8 TEST FOR SODIUM

8.1 Apparatus

A-8.1.1 *Flame Photometer* — equipped with interference filter.

NOTE — Sodium filter to be used should be of preselected or metal filter type, since ordinary sodium filters (for example Gelatin filters) have only 1 percent selectivity and work properly when the ratio of Na:K is 100:1, whereas in potassium carbonate and potassium hydroxide, the ratio is exactly reverse. Hence results with gelatin filter are erratic.

A-8.2 Reagent

A-8.2.1 *Standard Sodium Solution*

Weigh exactly 2.542 g of sodium chloride, dissolve in de-ionized water and dilute to 1 000 ml in a measuring flask. Dilute 10 ml of this solution to 1 000 ml. One ml of the diluted solution contains 0.01 mg of sodium (as Na).

A-8.3 Sample Solution

Dissolve exactly 10 g of the material in de-ionized water and dilute to 1 000 ml in a volumetric flask. Dilute 100 ml of this solution to 1 000-ml in a volumetric flask. Use this solution for galvanometer readings. If needed, further dilute the solution.

A-8.4 Calibration Graph

Take 10, 20, 40, 60 and 80 ml of standard sodium solution in different measuring flasks and dilute to 100 ml with de-ionized water. Each flask now contains 0.1, 0.2, 0.4, 0.6 and 0.8 mg of sodium per 100 ml. Use these diluted solutions to obtain corresponding galvanometer reading as given in the procedure (**A-8.5.2**) and plot the concentration against galvanometer reading in a rectangular coordination graph. Draw a smooth curve over the points which gives a calibration graph in the range 0.1 to 1.0 percent sodium.

A-8.5 Procedure

A-8.5.1 Use a flame photometer equipped with atomizer, burner, optical selective device consisting of reflectors, lenses and diaphragms; and measuring instrument consisting of a photocell, an amplifier and a sensitive galvanometer. The galvanometer scale ranges from 0 to 100 divisions which measures the intensity of the radiation transmitted by the element.

A-8.5.2 Insert the sodium filter corresponding to wave-length 767 NM light burner fed by illuminating gas (laboratory gas) and adjust the specified air pressure between 0.5 to 0.7 kg/cm^2 and maintain the control knob. First spray de-ionized water and adjust the pointer to zero in galvanometer scale by adjustment knob. Then spray the standard sodium solution and adjust the deflection to maximum (100) by using sensitivity control knob. Again spray de-ionized water to see that pointer comes to zero; then spray standard solution to indicate 100. Repeat till de-ionized water reads zero and standard solution reads 100 with the same adjustment during both the operations. Reading zero by de-ionized water and with the same adjustment 100 by standard solution, indicates that the instrument is now ready for measurement.

A-8.5.3 Without altering the earlier adjustment of the instrument, spray various diluted solution prepared in **A-8.4** and obtain a calibration graph in the range 0.1 to 1.0 percent sodium. After washing with de-ionized water, spray the sample solution and obtain the galvanometer reading. From the graph read out the corresponding concentration of sodium (say A).

A-8.6 Calculation

$$\text{Sodium (as Na), percent by mass} = \frac{100A}{M}$$

where

A = concentration of sodium corresponding to the galvanometer reading (*see* **A-8.5.3**), and

M = mass in g of the material taken for preparing the sample solution.

A-9 TEST FOR SULPHATE

A-9.1 Reagents

A-9.1.1 *Concentrated Hydrochloric Acid* — *See* IS 265.

A-9.1.2 *Barium Chloride Solution* — 12 percent (*m/v*).

A-9.2 Procedure

To the filtrate reserved in **A-5.1**, add 1 ml of concentrated hydrochloric acid and heat to boiling. Add 5 ml of barium chloride solution with stirring and allow to stand overnight. Filter the precipitate through sintered glass crucible (G No. 4), wash with hot water and dry at 105°C. Cool in a desiccator and weigh.

A-9.3 Calculation

$$\text{Sulphate as (SO}_4\text{), percent by mass} = \frac{41.15\,M_1}{M}$$

where

M_1 = mass in g of the precipitate, and

M = mass in g of the material taken for the test (*see* **A-5.1**).

A-10 TEST FOR MONO OR TRIBASIC PHOSPHATE

A-10.1 Reagents

A-10.1.1 *Thymol Blue Indicator* — Dissolve 0.1 g of Thymol Blue in 100 ml of methanol.

A-10.1.2 *Standard Hydrochloric Acid* — 1 N.

A-10.2 Procedure

Weigh accurately 3 g of the material in 50 ml water in a 250-ml conical flask. Add 3 drops of Thymol Blue indicator. A blue colouration is produced which changes to yellow (with a greenish tinge) by the addition of not more than 0.4 ml of standard 1 N-Hydrochloric Acid which indicates the presence of mono or tribasic phosphate.

ANNEX B

(*Clause* 5.1)

SAMPLING OF DIPOTASSIUM HYDROGEN PHOSPHATE FOR ELECTROLESS PLATING

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, storing, preparing and handling test samples, the following precautions shall be observed.

B-1.1 Samples shall not be taken at a place exposed to weather.

B-1.2 The samples shall be placed in suitable, dry and air-tight containers preferably with replaceable closure.

B-1.3 While opening a sample container, adequate care shall be taken to prevent foreign particles from falling into the material.

B-1.4 Each sample container shall be marked with full details of sampling, the date of sampling, the year of manufacture and the batch number, if any.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute of lot.

B-2.2 For ascertaining the conformity of the material in the lot to the requirements of the standard, samples shall be tested from each lot separately.

B-2.3 For number of containers to be selected from the lot shall depend on the size of the lot and shall be as follows:

Lot Size, N	*Sample Size, n*
Up to 50	3
51 to 100	4
101 to 300	5
301 to 500	7
501 and above	10

B-2.3.1 These containers shall be selected at random from the lot. In order to ensure randomness of selection, reference may be made to IS 4905. In case this standard is not readily available the following procedure may be adopted:

> 'Starting from any container in the lot, count them in one order as 1, 2, 3.... up to *r* and so on where *r* is the integral part of *N*/*n* (*N* and *n* being the lot size and the sample size respectively). Every *r*th container thus counted shall be withdrawn till the requisite number of containers is obtained.'

B-3 TEST SAMPLES AND REFEREE SAMPLES

B-3.1 From each of the containers selected according to **B-2.3** a small quantity of material shall be taken from different parts of the container. The quantity of material shall be taken from different parts of the container. The quantity of material so drawn from each container shall be sufficient to make triplicate determinations for all the requirements given in Table 1 of this standard.

B-3.2 Out of these portions, approximately equal quantities of material shall be taken and mixed thoroughly so as to form a composite sample of quantity sufficient to make triplicate determinations for all the characteristics to be tested on the composite sample. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.3 The remaining portion of the material from each selected container shall be divided into three equal parts each forming an individual sample. One set of individual samples representing the containers sample shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.4 All the individual samples and the composite samples shall be transferred to separate sample containers. All the containers shall be sealed air-tight and labelled with full identification particulars.

B-3.5 The referee test samples consisting of a composite and a set of individual samples shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the two.

B-4 NUMBER OF TESTS

4.1 Tests for the requirements prescribed in Table 1 shall be conducted on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 Test on the Composite Sample

The lot shall be considered to have passed in respect of the characteristics tested on the composite sample (*see* **B-4.1**) if all the test results on the composite sample satisfy requirements given in Table 1.

B-5.2 The lot shall be declared as conforming to the requirements of this standard if **B-5.1** is satisfied.

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

Inorganic Chemicals and Photographic Materials Sectional Committee, CHD 1

Organization	*Representative(s)*
Central Salt and Marine Chemicals Research Institute (CSMCRI), Bhavnagar	Dr P. K. Ghose (***Chairman***)
Alkali Manufacturers' Association of India, New Delhi	Dr Y. R. Singh
Armed Forces Film and Photo Division (Ministry of Defence), New Delhi	Representative
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(*Continued on page* 7)

(*Continued from page* 6)

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
Telephones : 323 01 31, 323 33 75, 323 94 02

Telegrams: Manaksanstha
(Common to all offices)

Regional Offices : Telephone

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg
NEW DELHI 110002
{ 323 76 17
323 38 41

Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Kankurgachi
KOLKATA 700 054
{ 337 84 99, 337 85 61
337 86 26, 337 91 20

Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160 022
{ 60 38 43
60 20 25

Southern : C. I. T. Campus, IV Cross Road, CHENNAI 600 113
{ 254 12 16, 254 14 42
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